Measuring Peel Adhesion of Coatings

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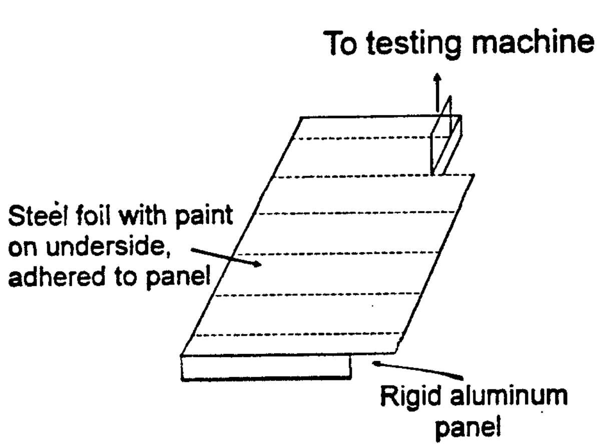
An essential requirement of protective coatings is that they adhere to the substrate to which they are applied. This is an obvious requirement, but it is a difficult one to measure quantitatively. Many procedures are available for assessing coating adhesion, including ASTM D 3359[1] for peel, ASTM D 4541[2] and ISO 4642[3] for tensile, and a reported procedure for shear.[4] However, all of the procedures have limitations. For example, the D 3359 peel test is qualitative, while the tensile and shear tests require gluing a loading fixture to the surface of the coating. None of the procedures is particularly suitable for determining adhesion of coatings exposed to a wet environment.

Fig. 1 - Schematic of test specimen used to measure peel strength of dry water-borne coatings. *Photo and figures courtesy of NIST*



This article describes a quantitative, repeatable procedure for measuring peel adhesion that was developed at the National Institute of Standards and Technology (NIST) primarily to measure the adhesion of coatings to a steel substrate exposed to a wet environment. Two examples of its application are presented. These examples are measurements of the wet adhesion strength of powder coatings to steel and the dry adhesion strength of water-borne coatings to steel.

NIST Peel Test Procedure

The NIST test was developed to quantitatively measure 90-degree peel adhesion of coatings. The test uses the peel test fixture, and a computerized universal testing machine (Fig. 2). The testing machine provides a constant rate of peel and continuously measures the force of detachment during the test. The peel fixture consists of a base plate bolted to the testing machine, a fixed slider (rail) attached to the base plate, and a moving slider (sled) that glides along the rail. Clamps on the sled hold a test panel in place. The low frictional force of the sled (about 2 N [0.4 lbf]) ensures that the peel angle of a coating strip remains at 90 degrees as the strip is peeled from the substrate. The peeling rates used in these tests were 20 mm/min (0.8 in./min) for the powder coating tests and 10 mm/min (0.4 in./min) for dry water-borne coatings.

A powder-coated test specimen is a typical coated laboratory test panel (e.g., hot rolled steel). The panel is first divided into strips by cutting through the coating to the substrate in several parallel lines along the length of the panel. One end of each strip is loosened to provide a tab for positioning in a grip of the testing machine.

The specimen for measuring the dry adhesion strength of the water-borne coatings is more complex. For this specimen, the coating is applied to a thin (250-micron [10-mil]) steel foil. In preparation for testing, the coated foil is cut into strips, and the coated side is glued to a rigid aluminum panel (Fig. 1).

Case One: Disbondment of Immersed Epoxy Powder Coatings on Steel

The peel strength adhesion of powder coatings applied to steel was measured as part of a project to study factors affecting the service life of powder-coated reinforcing steel in concrete structures exposed to marine immersion environments.[5] Peel strength adhesion measurements were made while the coating was wet because the adhesion of a coating to a steel substrate in a wet environment has been postulated as being a primary indicator of the corrosion control performance of a coating.[6] The information presented below on wet adhesion measurements summarizes the experimental procedure and results reported in reference 5.

The specimens used in the study were epoxy powder-coated, hot rolled steel panels that measured 100 mm x 150 mm x 3 mm (4 in. x 6 in. x 0.125 in.). The panels were abrasive blast cleaned (SSPC-SP 5, White Metal) to have a roughness of 50 to 75 μm (2 to 3 mils) before coating. The coating was applied in a powder coating facility to a dry film thickness (dft) of about 275 μm (11 mils). Each specimen was prepared by first placing a scribe perpendicular to the coating strips near one end of the panel measuring 60 mm x 0.25 mm (2.4 in. x 0.01 in.). The scribe was prepared using a diamond-tipped scoring device.

A plastic cell measuring 90 mm x 120 mm (3.5 in. x 4.7 in.) was cemented to the coated surface of the specimen to simulate a marine immersion environment. The cell was filled with a solution consisting of super-saturated $Ca(OH)_2$ and 3.5 percent NaCl in distilled water. The pH of the solution was maintained at 12, and the temperature was maintained at 35 C (95 F) throughout the test exposure. The solution was continuously aerated by bubbling air

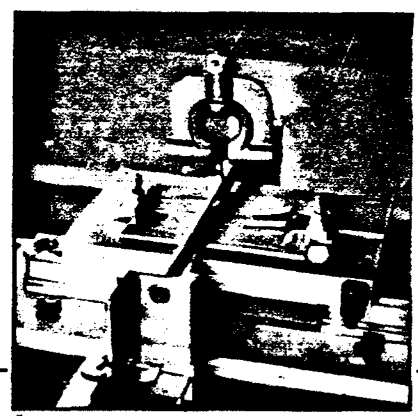
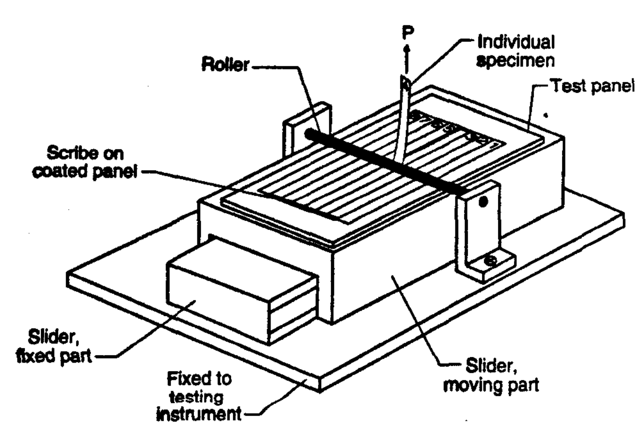


Fig. 2 - NIST test apparatus for measuring peel strength in the laboratory.



into the cell. Upon completion of the scheduled exposure time, a specimen was taken from the oven. The cell was then removed, and 8 strips were cut in the coating as shown in Fig. 2. The coating was tested within 30 minutes after the specimen was removed from the oven.

Wet adhesion peel strength results for a typical specimen immersed in the alkaline solution for 7 days are shown in Fig. 3. Adhesion peel strength is plotted for each of the 8 strips as a function of distance from the scribe. A primary feature of interest is the level of repeatability of results across and along the panel. The estimated coefficient of variation of the peel strengths of the 8 strips was 5 percent. The coefficient of variation is calculated by dividing the standard deviation of peel strengths by the mean peel strength. The high level of repeatability was also obtained in measurements of the wet peel strength of a powder coating applied to a smooth quartz substrate, for which the coefficient of variation was 3 percent.

Peel strength of a powder coating was measured as a function of immersion time in the alkaline solution to investigate relationships between coating performance and wet adhesion. The first major loss of adhesion appeared in a zone adjacent to the scribe as illustrated by the contour map shown in Fig. 4. In this zone, there was essentially no adhesion of the coating to the substrate. Using an independent chemical assessment procedure, it was determined that the zone was associated with cathodic delamination.[7]

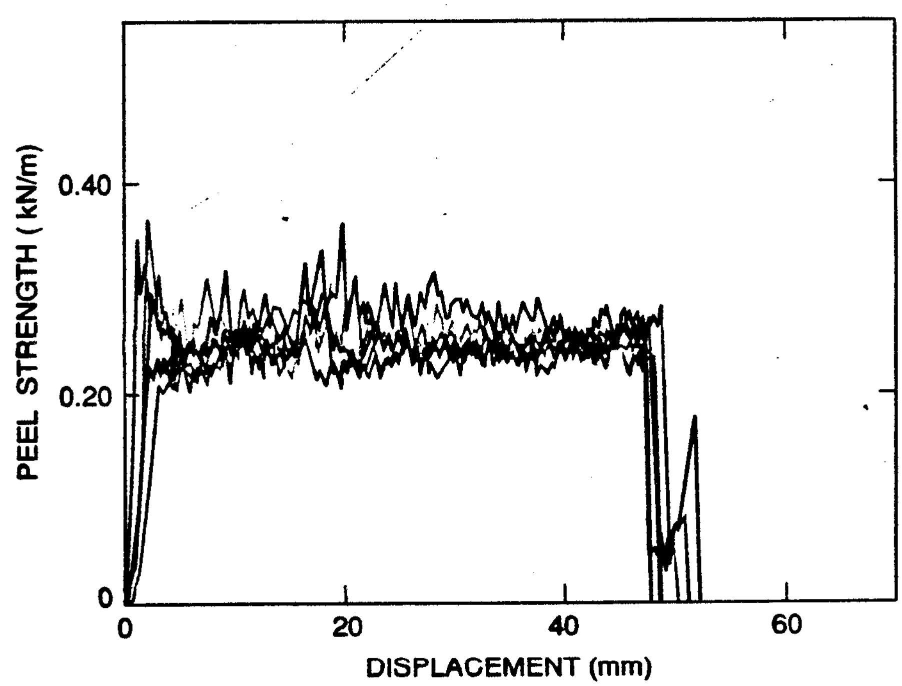


Fig. 3 - Typical peel strength curves of powder coating specimens peeled from a test panel after 7 days immersion.

Additional coated panels were evaluated for peel strength after varying immersion time periods. The cathodic delamination front moved at a nearly constant rate for about 50 days of immersion exposure, after which the rate slowed with longer immersion times (Fig. 6). Adhesion also decreased in the non-cathodic area with increasing immersion time. This is illustrated in Fig. 7 by the decreasing peel strengths away from the delaminated zone. For example, the peel strength after immersion for 22 days in the "plateau" region is less than the peel strength in that region after 17 days' immersion. Adhesion loss in this area (the wet adhesion reduction zone) was attributed to water accumulation at the interface.[7] The wet adhesion strength decreased for about 30 days and then remained nearly constant during the remainder of the 80-day exposure.

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The test procedure was also used to investigate the recovery of peel strength for a wet coating on a steel panel as a function of drying time at ambient laboratory condi-

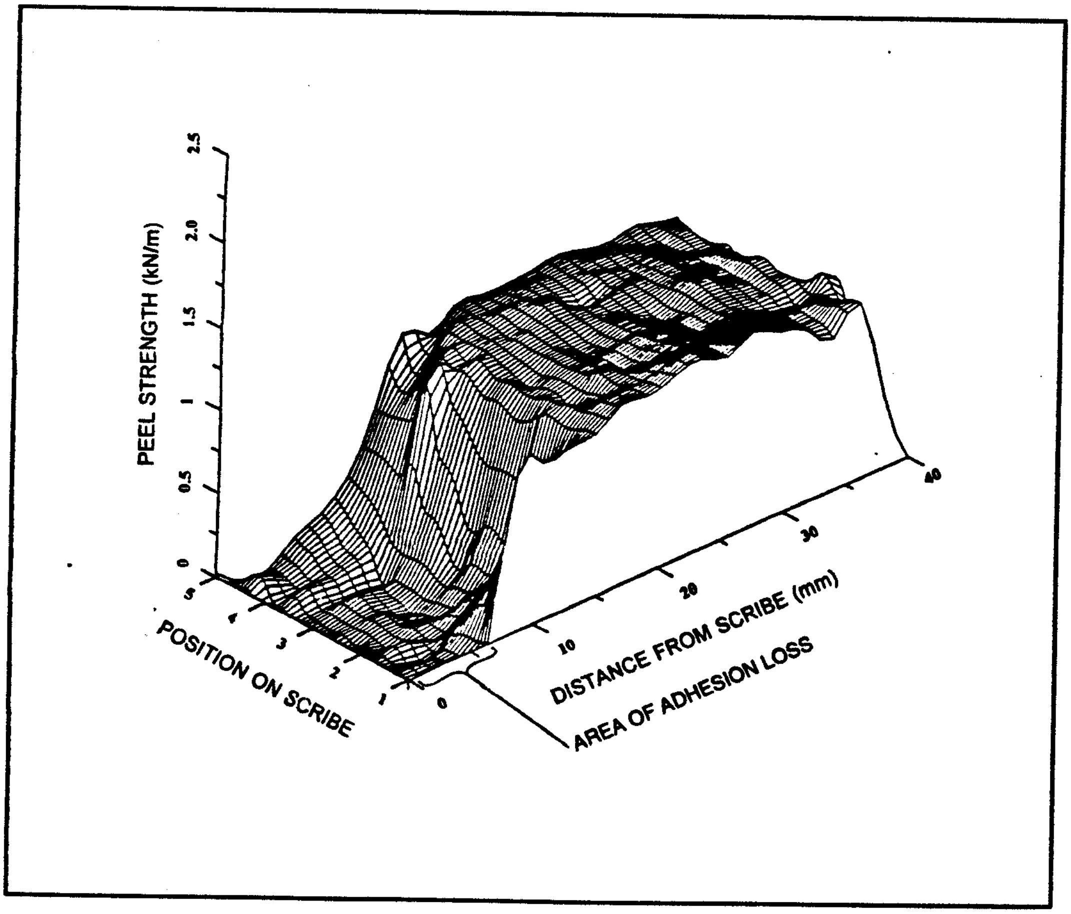

Fig. 4 - Three-dimensional contour map for wet peel strength of powder coatings after immersion for 22 days in the alkaline test solution, showing the cathodic delamination zone.

tions. Some recovery of peel strength was observed in the wet adhesion reduction zone after 12 days (Fig. 8). For example, the peel strength increased from 0.55 kN/m to about 0.73 kN/m when the immersed panel was allowed to dry for 8 days at ambient temperature before performing peel testing. However, no recovery was observed in the cathodic delamination zone.[7]

Case 2: Water-Borne Coatings

The use of the NIST peel procedure was investigated for specifying an adhesion criterion for encapsulant coatings. This criterion was to be used in a project to develop performance criteria for encapsulant coatings for lead-based paint. Because encapsulants are typically applied to aged lead-based paint films in residential buildings, a procedure for preparing standardized painted substrates representing the range of relevant properties of aged paint films was needed. These properties include chemical type, bulk integrity, adhesion to base substrate, and surface condition. Thus, water-borne and oil-based films that were prepared in the laboratory were desired. The films were to have either high or low cohesive strength, good or poor adhesion to the base substrate, and a clean or dirty surface. The peel adhesion results presented below were obtained in initial experiments to select 2 representative water-borne coatings that would fail cohesively at 2 widely different strengths.

The specimen (Fig. 1) was developed

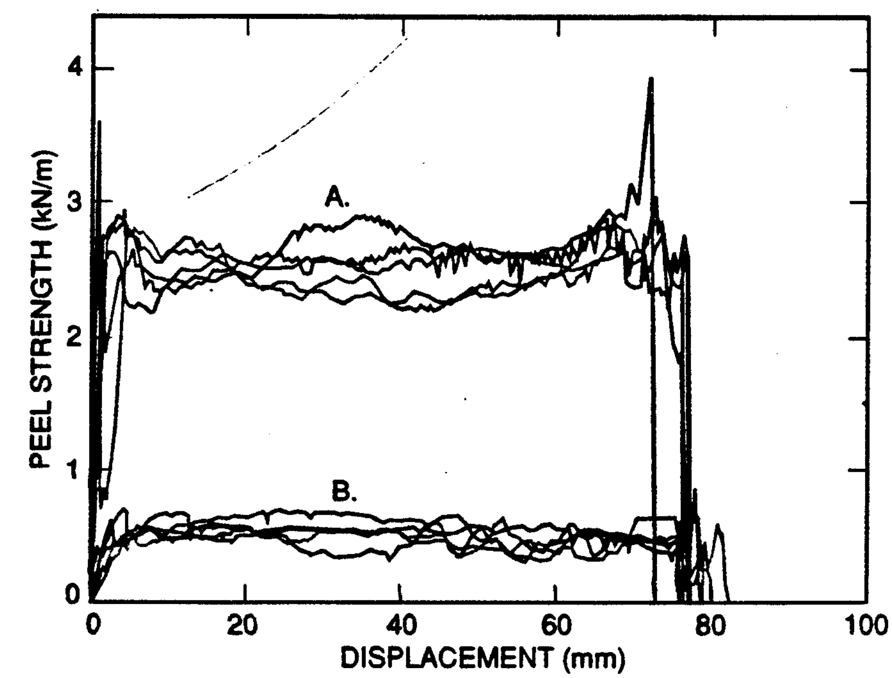

Fig. 5 - Typical peel strength test responses for modified specimens for a water-borne primer (A) and a water-borne finish coat (B). Each line represents the peel strength curve of an individual strip.

so that peel adhesion to a painted substrate could be measured for flexible as well as rigid encapsulant coatings. (An encapsulant coating will be sandwiched between the model coating and the aluminum panel, shown in Fig. 1.) In this specimen, the carbon-steel foil serves as both a substrate and a reinforcing material for the model coating film. The steel foil was prepared for coating by hot detergent cleaning and thorough rinsing in water. The model coatings were applied by drawdown to have a dft of about 75 μm (3 mils).

Coating peel strength was reduced on panels with flash rusting.

Several primers and finish waterborne coatings were tested to investigate adhesion and cohesive peel strengths. Peel strength results for the best adhering finish coat and primer are shown in Fig. 5. For both coatings, the peel failures were cohesive, with failure about midway through the thickness of the coating films. It should be noted that the cohesive strength of the primer was several times higher than that of the topcoat. The coefficient of variation of the peel strengths of the 5 strips making up a specimen was about 7 percent for the 2 coatings. The coefficient of variation between specimens was about 10 percent.

In the process of obtaining adequate quantities of the model coatings for the encapsulant project, 3 water-borne finish coatings formulated to meet the same specification were tested. In initial screening of the adhesion of these coatings to carbon steel, all were rated the same in a qualita-

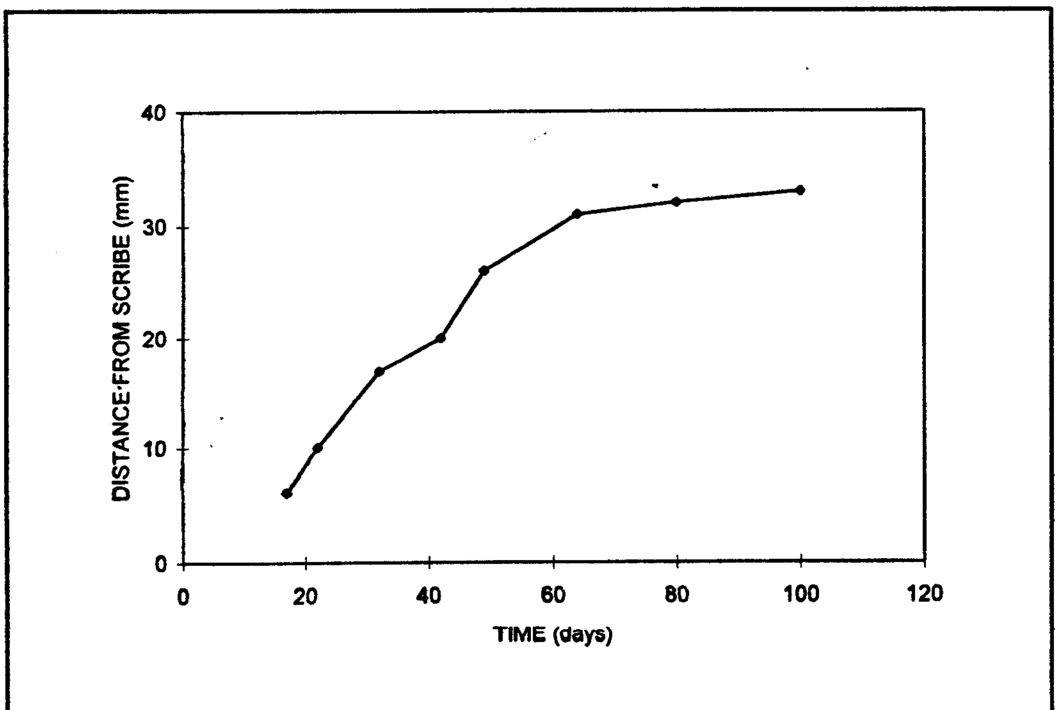

Fig. 6 - Distance of cathodic delamination from the scribe for a powder coating as a function of immersion time in the alkaline test solution.

tive knife test and ASTM D 3359 (i.e., 5B). However, when tested using the NIST procedure, one coating failed cohesively at about 2.0 kN/m (12 lbf/in.), while the other 2 failed adhesively at about 0.2 kN/m (1.2 lbf/in.).

The reasons for the differences in adhesion were not readily obvious. The nonvolatile vehicle and pigment contents were similar for all 3 coatings.

The coatings were oven dried to minimize flash rusting. Flash rusting was occasionally observed on specimens allowed to dry for several hours at room temperature. On specimens exhibiting flash rusting, coating peel strengths were greatly reduced as compared to those for specimens exhibiting no visible flash rusting.

Conclusions

The NIST procedure for conducting peel adhesion tests on coatings provides a method for obtaining quantitative, repeatable peel strength data. The coefficient of variation of measurements was less than 10 percent.

The method provides a means of obtaining additional insight into the mechanisms of failure of coatings applied to steel. In the powder coating study, 2 distinctive peel strength zones were easily detected for scribed coatings exposed to immersion environments.

The zones were associated with cathodic delamination near the scribe and with wet adhesion reduction further away from the scribe. Peel strength increased as the coating dried in the wet adhesion reduction zone but not in the cathodic delamination zone.

Significant differences in dry peel strengths were obtained for coatings that were given the same high adhesion rating when tested according to ASTM D 3359. These differences illustrate the superior sensitivity of the NIST peel procedure.

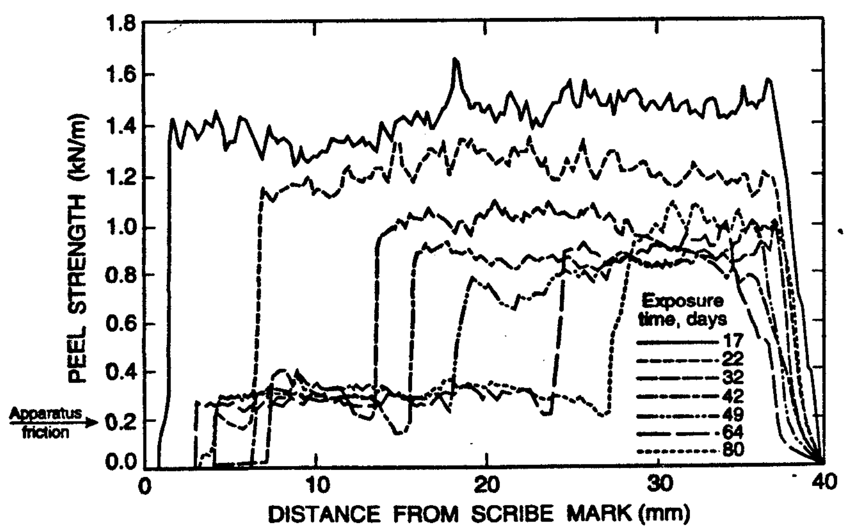

Fig. 7 - Peel strength curves of the center strips of powder coatings immersed for varying lengths of time.

For both wet and dry applications, this procedure should contribute to improved understanding of the adherence of coatings to steel and to improved coating formulations. JPCL

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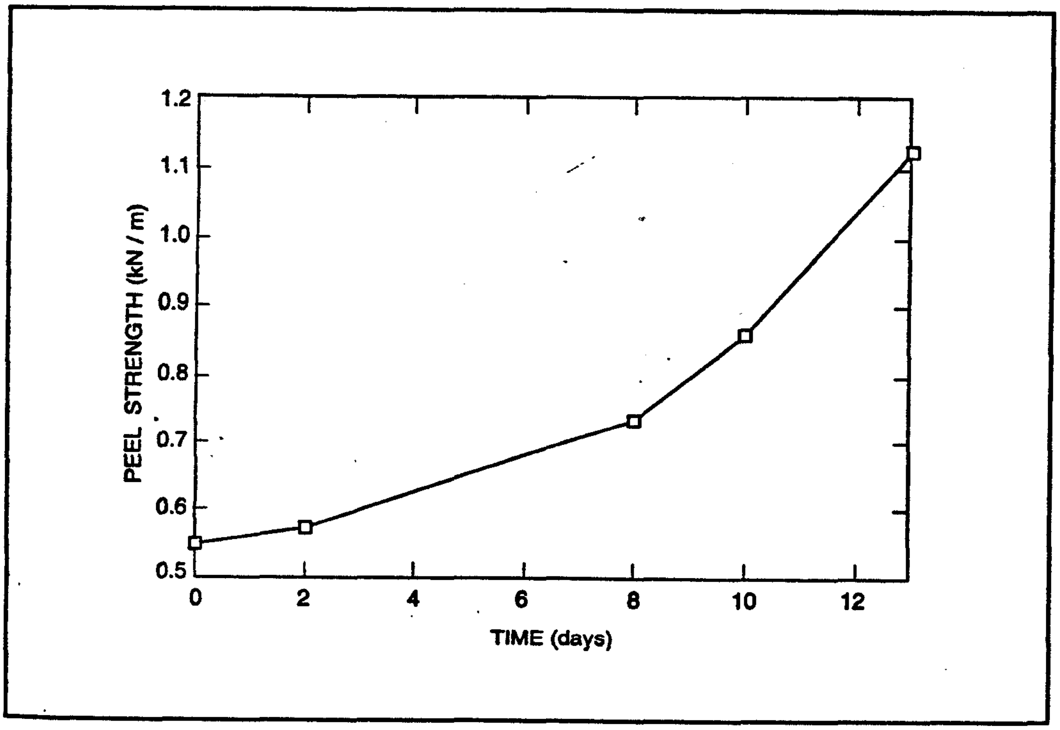

Fig. 8 - Peel strength of the center strip of a powder coating in the adhesion-reduction zone as a function of drying time at ambient conditions, showing extent of adhesion recovery.